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Events of Corrosion Phenomena on Carbon Steel Pipes in Environment of Sea Water and Ammonia Solutions due to the Presence of Sweet Gas

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Abstract. Based on the results of the study that the test sample material using carbon steel pipe plate API 5L-X65 is in chamber. With the research using the three point loading method is the environment of CO_2 gas and saturated H_2S gas in a solution of 7900 ml of sea water and 100 ml ammonia, the corrosion phenomenon occurs. And the corrosion event that occurs, is stress corrosion cracking transgranular and intergranular based the results of microstructure test results and based the results of polarized microscopy test. The corrosion rate that occurs will increase with the deflection given to the larger test samples for the same exposure time, the corrosion rate that occurs will increase with the stress σ given to the test sample getting larger for the same exposure time and inside crack will get deeper with the deflection given to the test sample getting larger for the same exposure time.
Keywords: stress corrosion cracking , sweet gas, specimen three-point loading.

1. Introduction

Research corrosion rate on carbon steel pipe API 5L-Grade B in the environment of H_2S gas CO_2 saturated condition in NaCl solution{1], the rate of corrosion in the carbon steel pipe 5L-Grade B in the H_2S gas atmosphere and the saturated CO_2 conditions in NaCl solution and acetic acid solution[2], study of crack corrosion cracking due to sweet gas (H_2S and CO_2) on carbon steel pipe API 5L-X65 to corrosion rate in acetic acid solution. [6] Characteristics of the Carbon steel Pipe 5L-X65 API with the Three Point Point H2S Gas Temperature Conditions Saturated CO_2 in Acetic Acid Solution[8], Hardness Testing and Tensile Test To Determine Hardening Coefficient n An API Material 5L-X65[7]. Analysis of corrosion phenomenon of carbon steel pipe plate API 5L-X65 in a solution of 250 ml of acetic acid and 4750 aquades under conditions of CO_2 and H_2S gas saturated at room temperature[15]. In the oil and gas industry in the event of stress corrosion cracking will result in fatal, ie pipe rupture that will result in oil and gas production will be stopped.

The corrosion rate on the 5L-X65 API carbon steel pipe with the three point loading method on acetic acid solution filled with CO_2 gas and H_2S in saturated state[13]. Analysis stress Corrosion Cracking on carbon steel pipe API 5L-X65 in Solution 7900

ml of Sea Water and 100 ml of Ammonia filled with CO_2 and H_2S Gases in Saturated Condition[14].

From the results of research on carbon steel pipe API 5L-X60 that is 17H1S will be susceptible to stresscorrosion cracking. And the mechanical properties of the degraded carbon steel pipe API 5L-X60 show higher resistance to corrosion cracking than in carbon steel pipes. Based on observations with fractographic that carbon steel pipe due to the hydrogen embrittlement process will occur stress corrosion cracking, that is caused by hydrogen atoms penetrating into the carbon steel pipe[11] .

High corrosion resistance in stainless steel carbon pipe (super martensite) is commonly used in the oil and gas industry, especially in acid environments. However there are some susceptible to the presence of hydrogen and the corrosion process mechanism will occur in the presence of H2S and depending on pH H2S diffuses seeping along the metal structure it will form sulfide metal pores as it reacts on the metal surface, thereby freeing the hydrogen bonded with sulfur which can absorbed and the hydrogen present in the H2S solution indirectly causes failure[12].

This results in the loss of early mechanical properties especially resistance to brittle fracture, which is based on engineering calculations at the pipe design stage. At the same time of stress corrosion cracking has been identified as one of the dominant failures in carbon steel pipes in humid environments, which would lead to the breakup of high-pressure gas transmission pipes as well as serious economic losses and disasters.[11] Commercial steel SS-430 undergoes a continuous corrosion process in the NaCl environment. The corrosion reaction that occurs, is dominated by an anodic reaction. The measured corrosion rate is very small with the tendency to decrease as the concentration of NaCl solution increases. So it can be concluded that SS-430 has outstanding corrosion resistance in NaCl environment. The corrosion products occurring on the SS-430 steel surface in the NaCl environment are predominantly chrome oxide and iron oxide. [4]

Based on the research that has been done, it can be seen that the effect of temperature on the protective properties of poly (TMSPMA) material on the surface of carbon steel by Electrochemical Impedance Spectroscopy (EIS) method in some temperature variations, 25ºC, 45ºC, 60ºC, and 75ºC. From the results of fittings made to the EIS measurement results obtained information that there is a decrease in the impedance value is often the temperature rise which shows that the resistance of corrosion protection material in critical condition to protect the carbon steel decreases. [5]

The corrosion occurs is the stress corrosion cracking transgranular and the stress corrosion crackin intergranular due to the sweet gas (CO2 and H2S gas) based on figure 7 and ammonia solution which is an element of ammonia anhydrous condensate. Crack deepness deeper with equal exposure time and variation stress σ based on figure 8. [15] Based on the experimental results it is shown that the dominant corrosion product formed is FeS and the corrosion resistance of tense crack increases with increasing workload, dissolved H2S concentration, and time of combustion.

2. Experimental Section

Tools and Materials

Tools and materials used in this research is a SEM-EDS, Optical Microscope , polarized microscope. The materials used in this research is the chamber, API 5L-X65 carbon steel plate, acetic acid (CH_3COOH), ammonia solution, aquadest, H_2S gas and CO_2 ga .

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Procedure

To make a house a corrosion test sample is required a sheet of steel in the form of a plate or flat extruded section with a rectangular cross section. In addition it can also be used cast iron or iron in the form of a beam as shown in Figure 1. The material to be tested is thinly sliced and then bent at both ends to obtain a voltage, and the magnitude of the voltage can be adjusted by providing a deflection through a screw-driven thread as shown in FIG. 1b. And this research uses 5L-X65 API carbon steel plate with length 12.5 cm, width 2 cm and thickness 2 mm stored in holder specimen three dot loading as shown (1.a) inserted into chamber room of corrosion test as much as 3 sample test corrosion with deflection variation as shown in figure (1.c) and image (1.d) is a 5L-X65 API test plate[3].

d. Samples of 5L-X6 API test specimens.

Figure 1. Holder shape and mounting of test specimen.

Research carried out that the carbon steel plate of API 5L-X65 is stored in the holder and then inserted into chamber chamber containing 4900 ml sea water and 100 ml ammonia as shown in Figure 2. The population of the corrosion test samples each time variation consists of three carbon API 5L API -X65 with three deflection variations, whereas the weight before and after the corrosion test was weighed.

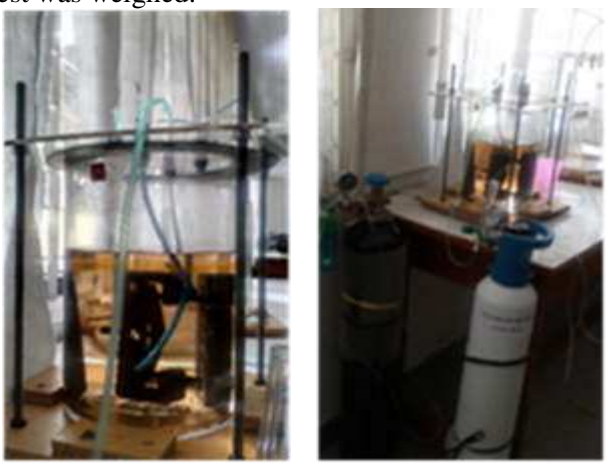

Figure 2. Chamber corrosion test.

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The tensile test is a mechanical stress-strain test that aims to determine the strength of the material to the tensile force as shown in figure 3. In the test that the test material is pulled to break and usually the focus is the maximum capability of the material in withstanding the tensile load, and the ability / strength This maximum pull is generally called "Ultimate Tensile Strength (UTS)". The long change in the curve is called the technical strain (εeng), which is defined as the change in length occurring due to a static change (ΔL) to the initial bar length (L0). The stress generated in this process is called the engineering voltage (σeng), which is defined as the loading value that occurs (F) on an initial cross-section (A0). The normal stresses due to static press charges can be determined based on the following equation:

$$\sigma = F/Ao \qquad (1)$$

with:

σ = Normal stress due to static tensile load (N / mm2).
F = Tensile load (N).
Ao = Area of initial specimen cross section (mm2) as shown in Figure 3.

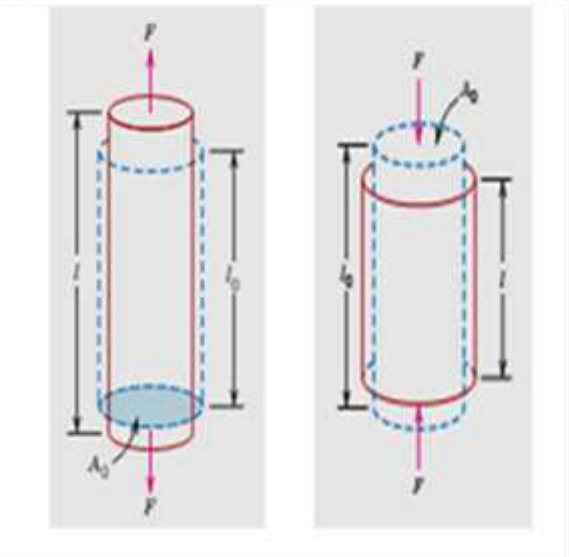

Figure 3. Basic principles of stress / stress (Callister, D. William 2007).

The strain due to the static tensile load can be determined by the following equation:

$$\varepsilon = \Delta L/L \qquad (2)$$

with:

ΔL = L-L0, ε = Strain due to static tensile load.
L = Change of length of specimen due to tensile load (mm).
Lo = The length of the initial specimen (mm).
Flat-shaped specimens have dimensions of width, length, and thickness of specimens usually determined by the product of the material used. The specimen holder in both ends of the specimen is bent / pressed with the screw (fitted with the ball), the support is in the middle of the plane. The dimensions of the specimens used can be modified according to

the specific needs (materials used), but their proportions are estimated proportionately. Calculation of elastic stress as in the following equation:

$$\sigma = 6 E t y/H2 \quad (3)$$

with:

σ = maximum tensile stress (N / m2).
E = modulus of elasticity (N / m2).
t = specimen thickness (mm).
y = maximum deflection (mm).
H = distance between external buffer (mm) and small deflection (y / H less than 0.1) as shown in Figure 3. Flat chip specimens with 25-51 mm (1-2 inches), 127-254 mm length (5 - 10 inches) and the thickness of the specimen is usually determined the product of the material used as shown in figure 1.

3. Results and Discussion

The data obtained based on the weight before and after the corrosion test was obtained to calculate the corrosion rate, and the test samples were then tested using microstructures and polarized microscope will be seen that the carbon steel plate image occurred corrosion and crack length. This research needs to be developed by using ultrasonic method to find out crack length that is based on reflection of wave that concerning surface of carbon steel plate. And the 5L-X65 API carbon steel plate in the chamber corrosion test chamber is loaded with saturated CO2 gas, and filled H2S gas for 10 minutes every two days as shown in table data 1.

Table 1. Corrosion Rate of Strained and Time Variations in Seeds 4700 ml, 100 ml Ammonia with H_2S and CO_2 Saturated At Room Temperature.

No.	$M_{initial}$ (gr)	M_{final}(gr)	PH initial	PH final	Length (cm)	Width (cm)	Thichness (cm)	Deflection (y) cm	Inside Crack initial (cm)	Time Exposure (hours)	Corrosion rate (mmpy)
A-1	52.99	52.96	14	10	13.1	2.42	2.4	0.5	0.02	336	0.031388956
A-2	53.73	53.53	14	10	13.1	2.42	2.4	1	0.02	336	0.209259709
A-3	53.18	52.69	14	10	13.1	2.42	2.4	1.5	0.02	336	0.512686287
B-1	53.21	52.76	14	9	13.1	2.42	2.4	0.5	0.02	672	0.235417172
B-2	53.29	52.65	14	9	13.1	2.42	2.4	1	0.02	672	0.334815534
B-3	53.96	52.76	14	9	13.1	2.42	2.4	1.5	0.02	672	0.627779127
C-1	53.33	52.95	14	9	13.1	2.42	2.4	0.5	0.02	1008	0.132531149
C-2	53.13	52.7	14	9	13.1	2.42	2.4	1	0.02	1008	0.149969458
C-3	53.26	52.63	14	9	13.1	2.42	2.4	1.5	0.02	1008	0.219722694

D-1	**53.35**	**53.21**	**14**	**9**	**13.1**	**2.42**	**2.4**	**1.5**	**0.02**	**1344**	**0.036620449**
D-2	**53.07**	**52.5**	**14**	**9**	**13.1**	**2.42**	**2.4**	**1**	**0.02**	**1344**	**0.149097543**
D-3	**53.84**	**52.66**	**14**	**9**	**13.1**	**2.42**	**2.4**	**0.5**	**0.02**	**1344**	**0.308658071**

The data in table 2 that the magnitude of the strain of each carbon steel sample test sample (figure 1.c with different deflection) is inserted in the chamber as shown in figure 2, and the results of the study based on the variation of time and deflection variation are obtained as in table 1. Based on the data in table 1 that the corrosion rate to the variation of exposure time of the test sample with the same deflection, then obtained the corrosion rate as shown in figure 3 is like a parabola. And the corrosion rate to the variation of stress σ at the same exposure time is obtained as in figure 4, ie the greater the stress σ given the greater the corrosion rate for the same time exposure.

Table 2. Table of calculation stress based on equation 3.

No	E (N/m²)	t (m)	y (m)	H (m)	H ² (m²)	σ (N/m²)
1	2.05E+11	0.0022	0.005	0.0946	0.008949	1.51E+09
2	2.05E+11	0.0022	0.01	0.0946	0.008949	3.02E+09
3	2.05E+11	0.0022	0.015	0.0946	0.008949	4.54E+09

Table 3. Table of calculation stress and Corrosion Rate (mmpy).

No.	Stress (N/m^2)	Corrosion rate (mmpy)
1	1511873740	0.031388956
2	3023747480	0.209259709
3	4535621220	0.512686287
1	1511873740	0.235417172
2	3023747480	0.334815534
3	4535621220	0.627779127
1	1511873740	0.132531149
2	3023747480	0.149969458
3	4535621220	0.219722694
1	1511873740	0.036620449
2	3023747480	0.149097543
3	4535621220	0.308658071

Table 4. Data on exposure time and depth of crack.

No.	Time exposure (jam)	Depth Crack (µm)

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1	336	62.36
2	672	65.32
3	1008	66.98
4	1344	67.75
1	336	69.93
2	672	76.76
3	1008	78.94
4	1344	79.89
1	336	85.06
2	672	89.86
3	1008	90.79
4	1344	116.99

Based on table 1 and figure 3, the corrosion rate at the same exposure time will increase as the deflection is given to the test sample, ie the corrosion rate is $\mathbf{y = 0.2511x^2 - 0.0209x - 0.0209}$ for exposure time 336 hours , $\mathbf{y = 0.3871x^2 - 0.3819x + 0.3296}$ for exposure time 672 hours, $\mathbf{y = 0.1046x^2 - 0.1221x + 0.1674}$ for exposure time 1008 hours and $\mathbf{y = 0.0942x^2 + 0.0837x - 0.0288}$ for exposure time 1344 hours.

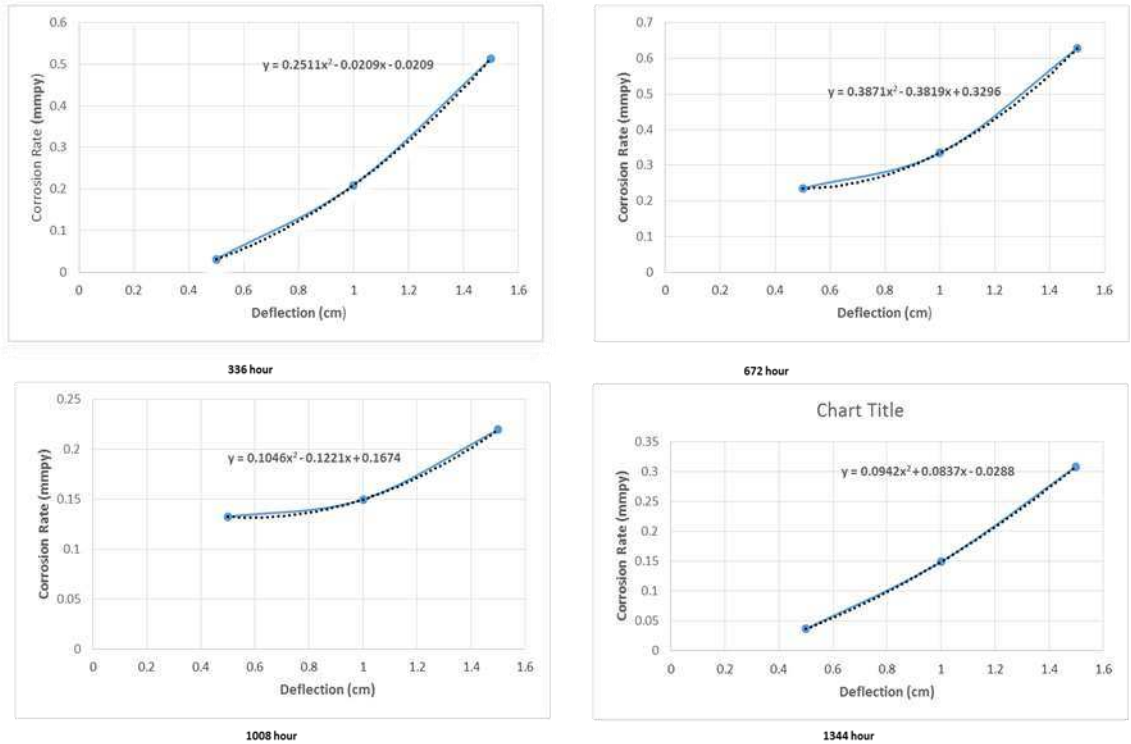

Figure 3. Graph of corrosion rate against deflection variation at the same time exposure.

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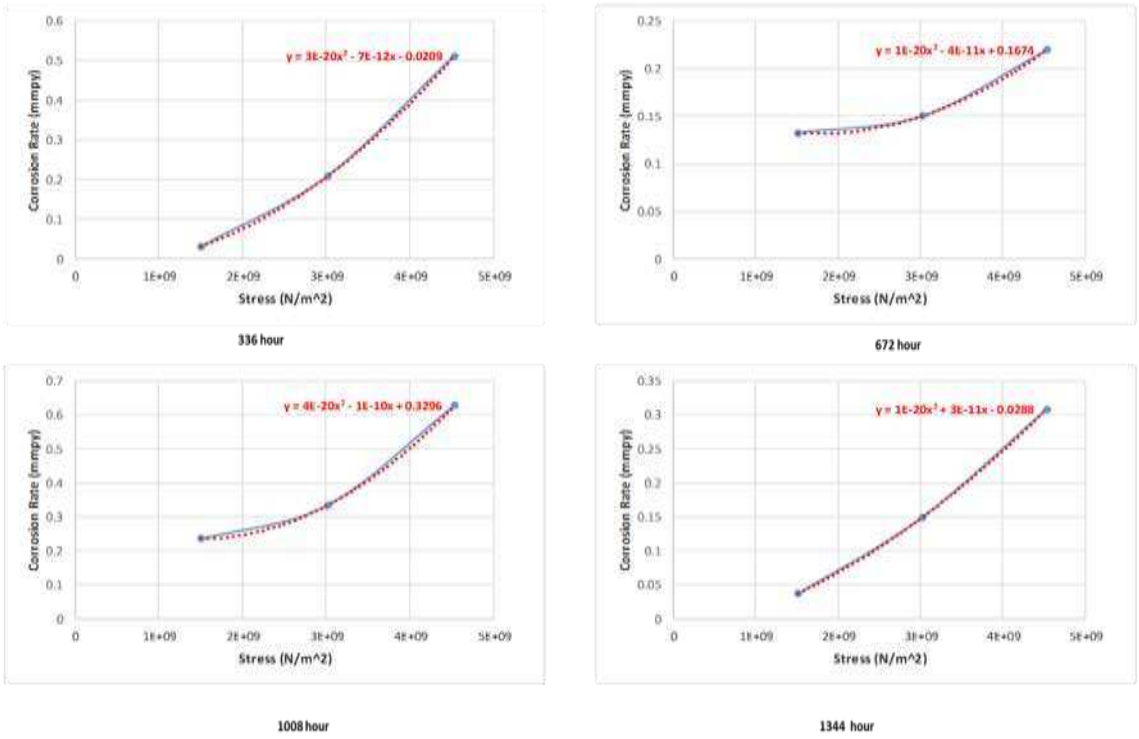

Figure 4. Graph of Corrosion Rate on the variation of stress σ with the same time exposure.

Based on table 4 that the depth crack will increase in time as the exposure time is given to the test sample as shown in figure 5, and based on result microstructure shown as in figure 8 with the depth crack **y = -5E-06x^2 + 0.0135x + 58.407** for 0.5 cm deflection, **y = -1E-05x^2 + 0.0314 + 61.015** for deflection 1.0 cm and **y = 5E-0.5x^2 – 0.0508x + 98.245** for deflection 1.5 cm.

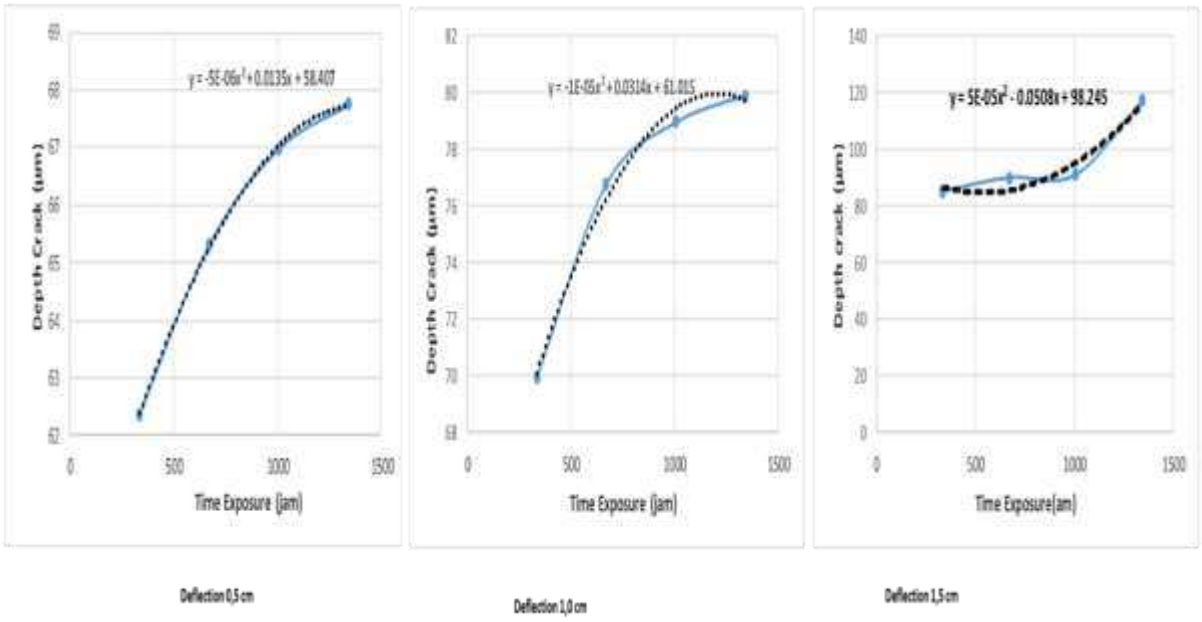

Figure 5. Depth Crack to exposure time with the same deflection.

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Based on the result of microstructure as shown in figure 6 that the uhi sample which is in the solution of 7900 ml sea water and 100 ml ammonia after filled H_2S gas and CO_2 gas happened corrosion phenomenon, ie stress corrosion cracking of transgranular and stress crack corrosion of intergranular. And based on the results of polarized microscope as in figure 7 there is event a stress corrosion cracking by comparing the results of the microstructure for the same sample.

Figure 6. Results of microstructure.

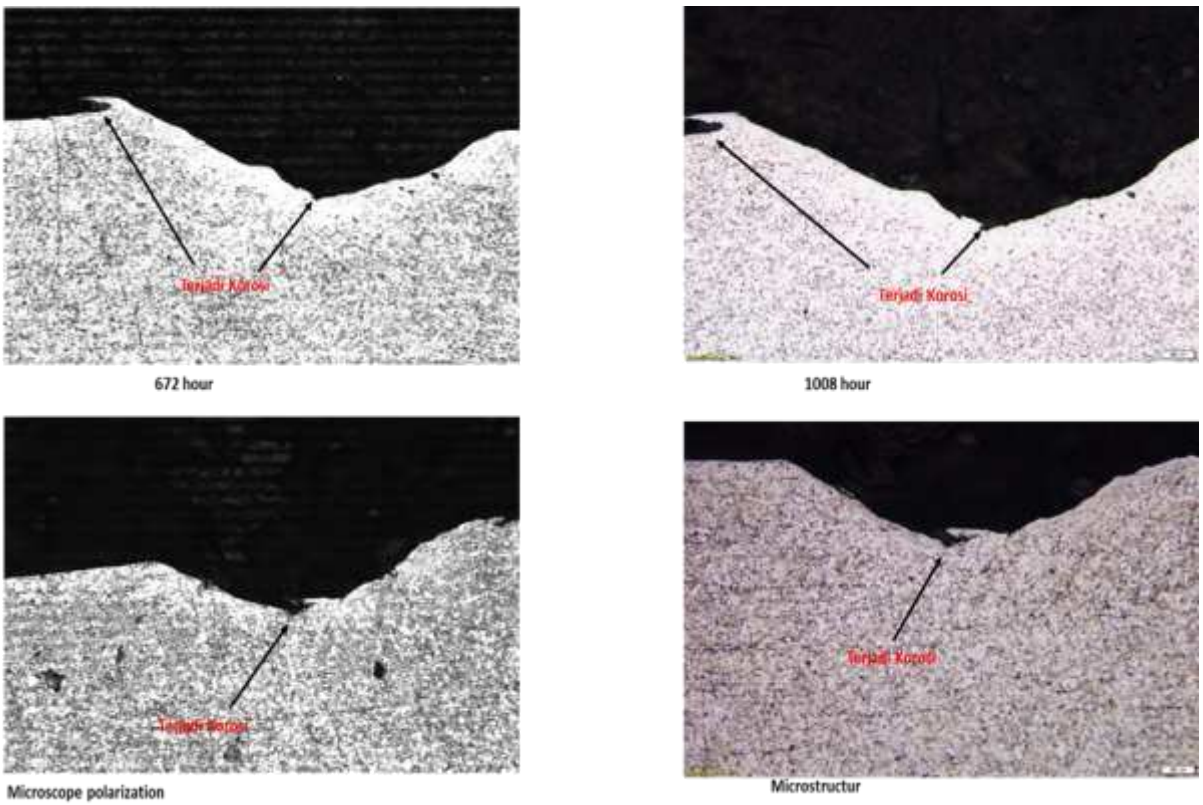

Figure 7. Results of microstructures and polarized microscopy results for the same test sample.

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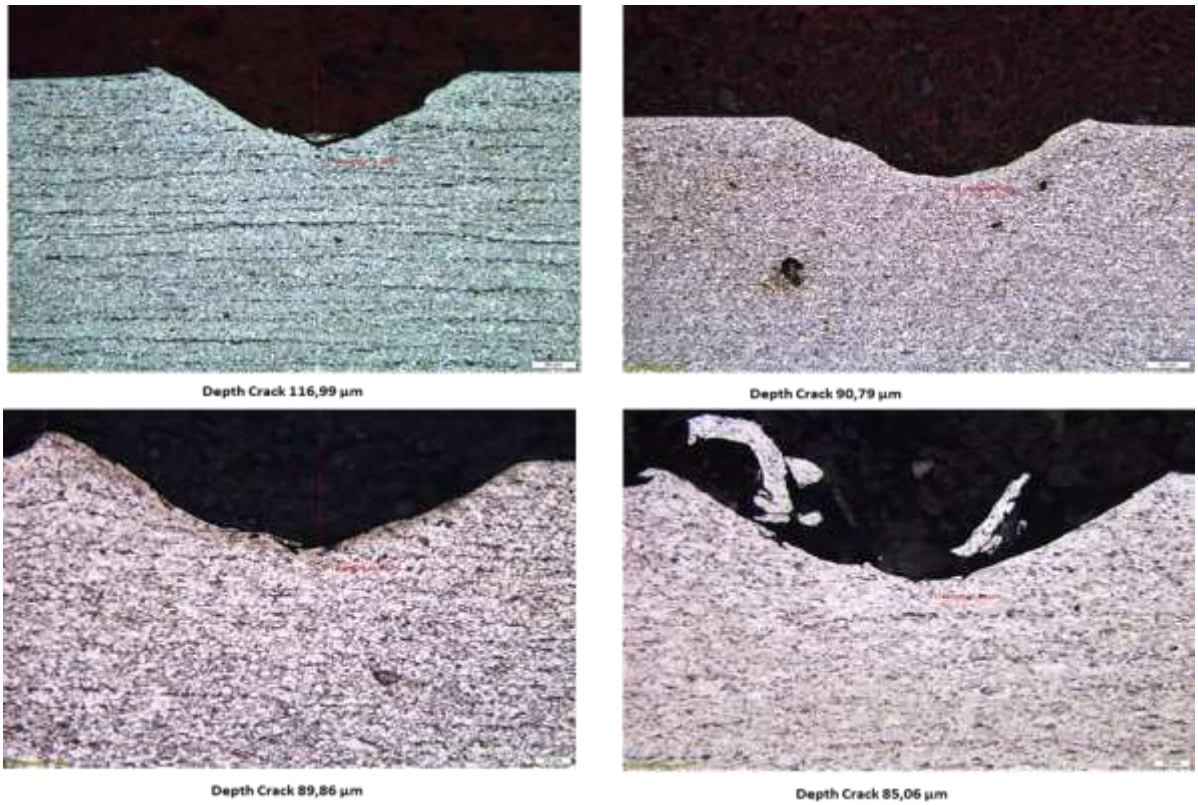

Figure 8. Depth crack based on microstructure results.

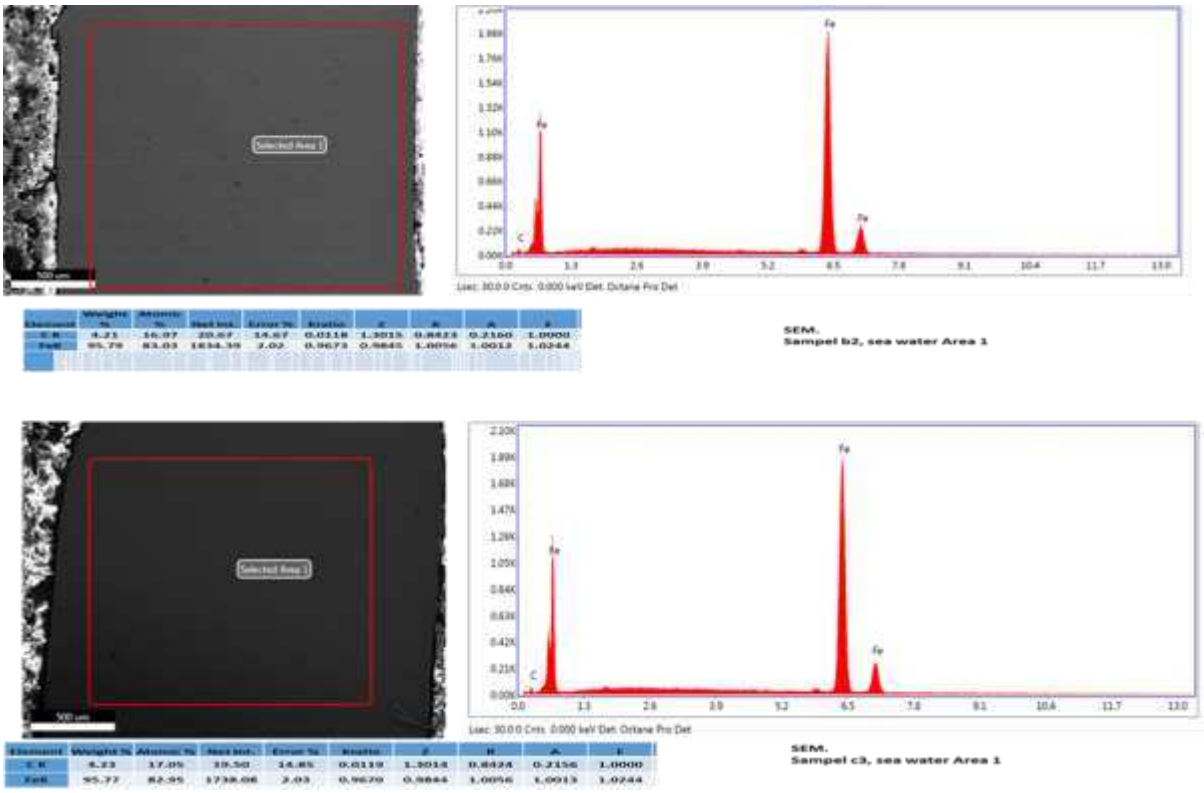

Figure 9. Result SEM-EDS for test sample b2 and c3 test sample.

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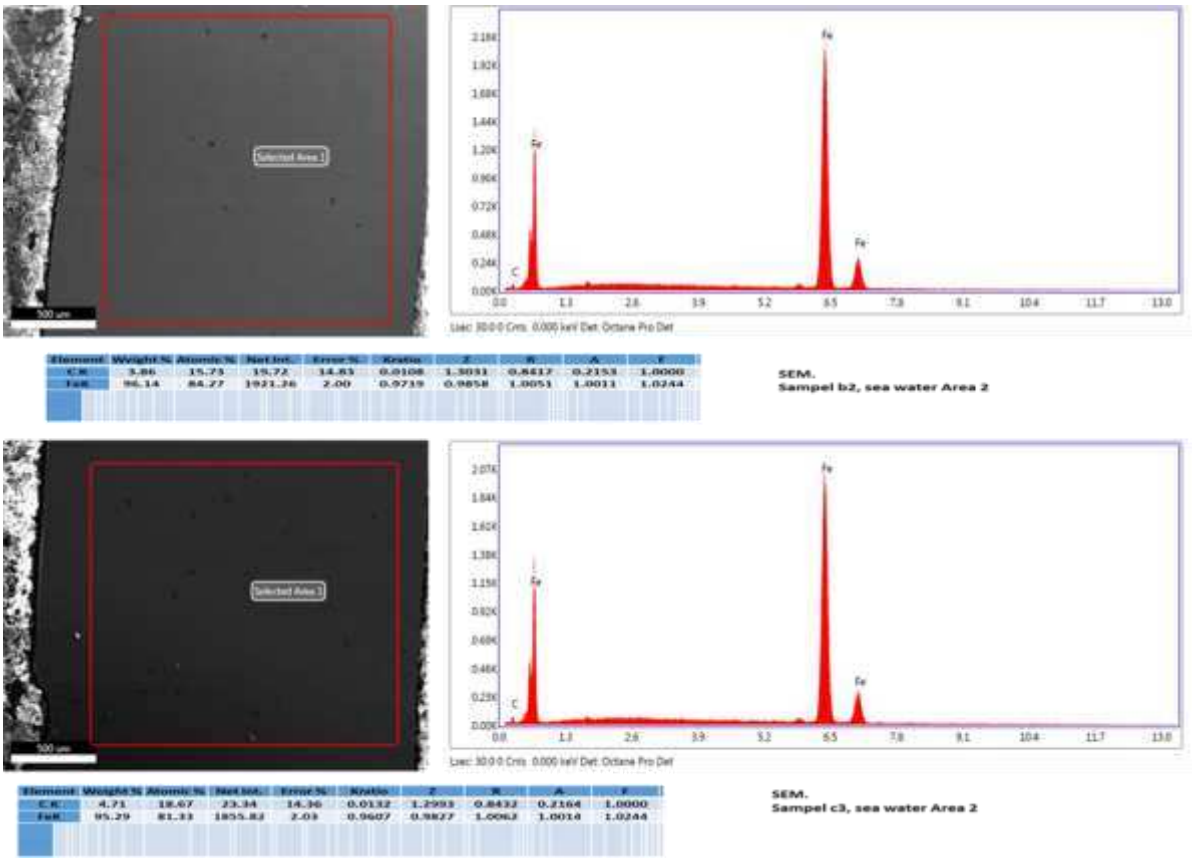

Figure 10. Result SEM-EDS for b2 and c3 samples.

Table 4. percentage weight and percentage atoms for element C and element Fe

No.	Weight C (%)	Atomic C (%)	Weight Fe (%)	Atomic Fe (%)
1	4.21	16.97	95.79	83.03
2	4.23	17.05	95.77	83.95
3	3.86	15.73	96.14	84.27
4	4.71	18.67	95.29	81.33
average	4.2525	17.105	95.7475	83.145

The result of the SEM-EDS test as shown in Figure 9 for the B2 test sample that the C element has a weight of 4.21% and the atomic composition is 16.97%, while Fe element has a weight of 95.79% and the atom composition is 83.03%. And SEM-EDS test result for C3 sample that element C has 4.23% weight and atom composition 17,05%, while element Fe has 95,77% weight and atom composition 83,95%. Based on Figure 10 for the test sample B2 that element C has a weight of 3.86% and the atomic composition is 15.73%, while Fe element has a weight of 96.14% and the atomic composition is 84.27%. And the SEM-EDS test results for C3 test assays that element C has a weight of 4.71% and its atomic composition is 18.67%, while Fe element has a

weight of 95.29% and its atomic composition is 81.33%. So the average composition for element C is that it weighs 4.2525%, the atom is 17.105% and for element Fe is that a weighs 95.77475%, the atom is 83.145%.

Based on the data as in Table 1 and based on the graph as shown in FIG. 3 that the corrosion rate increases with the amount of deflection given to the test sample, and the table 3 of figure 4 corrosion rate increases with the magnitude of the strain σ given to the test sample. And the test samples which are in the solution of 7900 ml of sea water and 100 ml of ammonia with loaded CO2 gas in saturated state and H2S gas every two days for 10 minutes then there is corrosion crack voltage (SCC) because CO2 gas, H2S gas and ammonia are source corrosive.

The corrosion events occur are the stress corrosion cracking transgranular and intergranular as shown in figure 6, and based on the results of polarized microscopes as in figure 7 also occurred corrosion events. And percentage component of the chemical element that is dominant based on the results of the SEM-EDS test for sample b2 and sample c3 as shown in the table 4, which is either the percentage of the weight or percentage of the atom.

4. Conclusion

Synthesis of copper oxide on copper thin plate (thickness of 200-250 μm) was successfully performed at temperature 380oC for 1 hour. The characterization results of DTA, SEM and XRD have provided information that during thermal oxidation formed 92.6% Cu_2O and 6.4% CuO with 25 μm thickness and maximum oxygen attack at 129 μm depth. Oxides formed on the oxidation process can be applied to visible light photovoltaic panels with continued research of physical properties and optical materials to obtain materials with appropriate photovoltaic functions.

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